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IS 14780 (2000): Zinc Naphthenate [CHD 1: Inorganic Chemicals]

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भारतीय मानक

# ज़िंक नेफ्थिनेट — विशिष्टि

*Indian Standard*

# ZINC NAPHTHENATE — SPECIFICATION

ICS 71.060.01

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**BUREAU OF INDIAN STANDARDS**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*August* 2000

**Price Group 4**

General Inorganic Chemicals Sectional Committee, CHD 3

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Zinc naphthenate has the molecular formula $Zn[(CH_2)_5COOH]_2$. It is insoluble in water but soluble in organic acids.

Zinc naphthenate is used as paint film improver and the automobile primer coatings. Naphthenates are well known for their fungicidal activity. They provide long term protection against fungus, mold, mildew and marine parasites. They also protect canvas, burlap, tents, lumber and utility poles. Zinc naphthenate is also used as a preservative.

There is no ISO specification for this subject.

Composition of the Committee responsible for formulating this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# *Indian Standard*

# ZINC NAPHTHENATE — SPECIFICATION

## 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for zinc naphthenate.

## 2 NORMATIVE REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provision of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

| *IS No.* | *Title* |
|---|---|
| 265 : 1993 | Hydrochloric acid (*fourth revision*) |
| 460 : 1985 | Test sieves (*third revision*) |
| 1070 : 1992 | Reagent grade water (*third revision*) |
| 1078 : 1987 | Copper naphthenate (*second revision*) |
| 9252 : 1979 | Method for determination of zinc (volumetric) |

## 3 REQUIREMENTS

### 3.1 Description

The material shall be the zinc salt of cycloparaffin carboxylic acids extracted from mineral oils and shall be in the form of a hard solid or liquid of amber colour and shall not contain any solvent.

**3.2** The material shall also comply with the requirements prescribed in Table 1, when tested according to the methods prescribed in Annex A and IS 1078. Reference to the relevant clauses of Annex A of IS 1078 is given in col 4 and 5 of Table 1.

### 3.3 Requirements of Extracted Naphthenic Acid from Zinc Naphthenate

The requirements of naphthenic acid extracted from zinc naphthenate by the method prescribed in **A-8** of IS 1078 shall comply with the requirements prescribed in Table 2, when tested according to the methods prescribed in Annex B of IS 1078. Reference to the relevant clauses of Annex B of IS 1078 is given in col 4 of Table 2.

**Table 1 Requirements for Zinc Naphthenate**
(*Clause* 3.2)

| Sl No. | Characteristic | Requirement | Method of Test, Ref to | |
|---|---|---|---|---|
| | | | Annex | IS 1078 |
| (1) | (2) | (3) | (4) | (5) |
| i) | Zinc (as Zn), percent by mass, *Min* | 14.0 | A | — |
| ii) | Matter insoluble in acetone, percent by mass, *Max* | 1.0 | — | A-3 |
| iii) | Mineral acidity | To pass the test | — | A-4 |
| iv) | Chlorides (as NaCl), percent by mass, *Max* | 0.1 | — | A-5 |
| v) | Sulphates (as $Na_2SO_4$), percent by mass, *Max* | 0.15 | — | A-6 |
| vi) | Sulphonic acids and their derivatives (as $Na_2SO_4$), percent by mass, *Max* | 1.5 | — | A-7 |

**Table 2 Requirements for Naphthenic Acid**
(*Clause* 3.3)

| Sl No. | Characteristic | Requirement | Method of Test, Ref to IS 1078 |
|---|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | Acid value | 170 - 240 | B-1 |
| ii) | Iodine value, *Max* | 2.0 | B-2 |
| iii) | Relative density | 0.95 | B-3 |
| iv) | Refractive index | 1.45 - 1.47 | B-4 |

## 4 PACKING AND MARKING

### 4.1 Packing

The material shall be packed in containers as agreed to between the purchaser and the suppliers.

### 4.2 Marking

The containers shall be securely closed and marked with the following information:

a) Name and net mass of the material;
b) Indication of the source of manufacture;
c) Month and year of manufacture; and
d) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

#### 4.2.1 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

**4.2.1.1** The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## 5 SAMPLING AND CRITERIA FOR CONFORMITY

The method of drawing representative samples of the material from a lot, number of tests to be performed and the method of finding out the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex C of IS 1078.

## 6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

# ANNEX A

[*Table* 1, *Sl No.* (i)]

## DETERMINATION OF ZINC

## A-1 GENERAL

Two methods are prescribed, namely, potassium ferrocyanide method (Method A) and ethylene-diamine tetracetate (EDTA) method (Method B). In case of dispute, Method B should be the referee method.

## A-2 METHOD A (POTASSIUM FERROCYANIDE METHOD)

### A-2.1 Outline of the Method

The sample is decomposed with hydrochloric acid and nitric acid and hydrogen sulphide gas is passed to precipitate cadmium and copper. Ammonium chloride, ammonium hydroxide and bromine water are added to the filtrate to remove iron and manganese. Hydrochloric acid is added to the solution which is titrated with potassium ferrocyanide standard solution using uranyl salt indicator.

### A-2.2 Reagents

**A-2.2.1** *Concentrated Hydrochloric Acid*

See IS 265.

**A-2.2.2** *Hydrochloric Acid*

1:1, 1:2, 1:5 and 1:50 (*v*/*v*).

**A-2.2.3** *Nitric Acid*

1:1 (*v*/*v*).

**A-2.2.4** *Sulphuric Acid*

1:2 (*v*/*v*).

**A-2.2.5** *Ammonium Hydroxide*

**A-2.2.6** *Bromine Water*

**A-2.2.7** *Hydrogen Peroxide*

1:10 (*v*/*v*).

**A-2.2.8** *Ammonium Chloride*

**A-2.2.9** *Ammonium Persulphate*

**A-2.2.10** *Ammonium Carbonate*

**A-2.2.11** *Ammonium Chloride Wash Solution*

Dissolve 20 g of ammonium chloride in water, add 10 ml of ammonium hydroxide and dilute to 1 litre.

**A-2.2.12** *Litmus Paper*

**A-2.2.13** *Hydrogen Sulphide Water*

Saturated.

**A-2.2.14** *Uranyl Salt Indicator*

Dissolve 10 g of uranyl acetate or uranyl nitrate in water and dilute to 100 ml.

**A-2.2.15** *Potassium Ferrocyanide Standard Solution*

Dissolve 21.6 g of potassium ferrocyanide in water, and dilute with water to 1 litre. Standardize this solution as follows:

> Weigh accurately 0.25 g of zinc into a 500-ml conical flask. Add 10 ml of hydrochloric acid to dissolve it completely. Then add 100 ml of water and 14 g of ammonium chloride and neutralize with ammonium hydroxide using litmus paper and add 5 ml of hydrochloric acid. Add 50 ml of hydrogen sulphide water (saturated) and dilute with water to about 250 ml. Titrate zinc in accordance with

**A-2.3.8** and calculate the mass of zinc equivalent to 1 ml of potassium ferrocyanide solution as follows;

$$m = \frac{M}{V_1 - V_2}$$

where

$m$ = mass in g of zinc equivalent to 1 ml of potassium ferrocyanide solution,

$M$ = mass in g of zinc taken,

$V_1$ = volume in ml of potassium ferrocyanide solution consumed in titration, and

$V_2$ = volume in ml of potassium ferrocyanide solution consumed for the blank.

Store this standard solution in a brown bottle in a dark place and restandardize prior to use.

### A-2.3 Procedure

**A-2.3.1** Weigh accurately 0.3 to 1 g of the sample into a 300-ml beaker. Cover with a watch glass and moisten with small amount of water. Add 20 to 30 ml of hydrochloric acid and gently heat to volatilize sulphur contained in sulphide as hydrogen sulphide gas. Add 10 ml of nitric acid continue the heating to decompose the sample completely and evaporate nearly to dryness. After cooling, add 10 ml of hydrochloric acid (1:1) to dissolve soluble salts and dilute with water to 3 percent by volume of hydrochloric acid solution.

**A-2.3.2** Heat the solution to 80 to 90°C and immediately pass hydrogen sulphide gas for 20 to 30 minutes. After standing a while, filter and wash the filter paper with hydrochloric acid (1:50) saturated with hydrogen sulphide gas collect the filtrate and washings in a 500-ml beaker and reserve as the main solution.

**A-2.3.3** Place the original beaker under the funnel, add dropwise 9 ml of hydrochloric acid (1:2) into the filter paper to dissolve the precipitate and wash with about 100 ml of warm water.

**A-2.3.4** Heat the solution and pass hydrogen sulphide gas to precipitate sulphides in the same way as prescribed in **A-2.3.2**. Filter, wash and combine the filtrate and washings with the main solution reserved in **A-2.3.2**.

**A-2.3.5** Heat the solution to evaporate to about 50 ml and add a small amount of bromide water or hydrogen peroxide water. Heat the solution to oxidize iron, etc, and add 5 g of ammonium chloride. Add, under stirring, 15 ml of ammonium hydroxide to precipitate iron, etc, followed by 10 to 20 ml of bromine water or 0.5 g of ammonium persulphate, and boil the solution for a few minutes covered with a watch glass.

**A-2.3.6** After standing a while, filter the solution into a 500-ml conical flask and wash the filter paper several times with ammonium chloride wash solution. Reserve the filtrate and washings as the main solution. Wash the precipitate with water into the original beaker, add 10 ml of hydrochloric acid (1:1) and heat to dissolve the precipitate.

**A-2.3.7** Add 3 g of ammonium chloride and repeat the procedure as in **A-2.3.5** with the addition to ammonium hydroxide and bromine water or ammonium persulphate to precipitate iron, etc. Filter the precipitate through the original filter paper and wash with ammonium chloride wash solution. Combine the filtrate and washings with the main solution. Boil the solution for a few minutes to decompose the excess of bromine or ammonium persulphate present and adjust the volume of the solution to about 200 ml.

**A-2.3.8** Add dropwise hydrochloric acid to neutralize the solution agent litmus paper. Add additional 5 ml of the acid followed by 50 ml of hydrogen sulphide water (saturated) and titrate the solution with potassium ferrocyanide solution using uranyl salt as the external indicator keeping the solution temperature at 75 to 80°C. In the titration, add potassium ferrocyanide solution in small increments at first and shake the flask every time. The precipitate produced will gradually become fine as the titration proceeds and becomes milky at the approach of the end point. Add 1 or 2 drops of the standard solution and shake the flask after each addition. Take one drop of the solution with a glass rod on a porcelain plate and mix with the external indicator placed on the plate. Continue the titration until the mixed solution changes colour from yellowish green to pale brown. Titrate three times to arrive at the exact volume of potassium ferrocyanide solution required. Run the blank for all reagents under the same condition and calculate the zinc content.

### A-2.4 Calculation

$$\text{Zinc (as Zn), percent by mass} = \frac{m \times (V_1 - V_2)}{M} \times 100$$

where

$m$ = equivalent in g of zinc to 1 ml of potassium ferrocyanide solution,

$V_1$ = volume in ml of potassium ferrocyanide solution consumed in titration,

$V_2$ = volume in ml of potassium ferrocyanide solution for the blank, and

$M$ = mass in g of the sample taken for the test solution.

## A-3 METHOD B (EDTA METHOD)

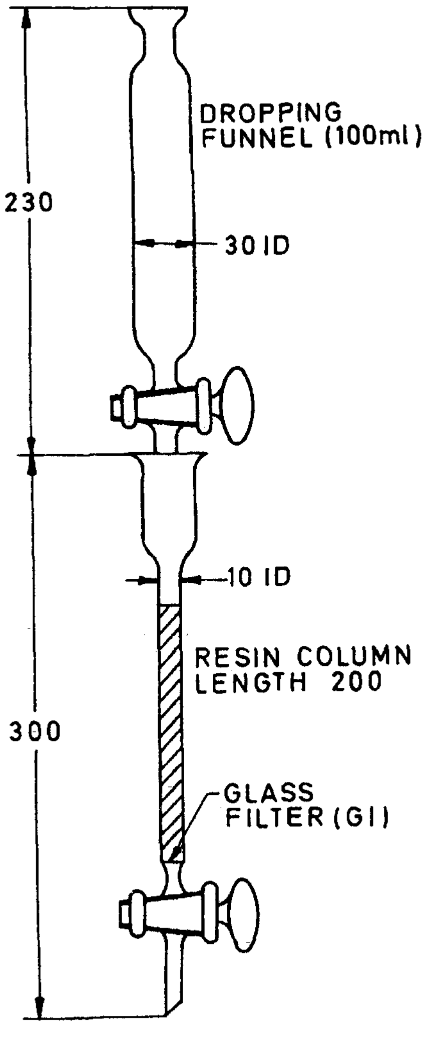
### A-3.1 Outline of the Method

The sample is decomposed with hydrochloric acid and nitric acid and zinc is retained on a strongly basic anion exchange resin. The resin column is washed with dilute hydrochloric acid and then zinc is eluted with dilute ammonium hydroxide containing ammonium chloride. The *p*H value of the elute is adjusted to about 5.5 and titrated with EDTA standard solution using xylenol orange indicator.

### A-3.2 Apparatus

**A-3.2.1** *Ion Exchange Resin Column*

Ion exchange resin column shall be as shown in Fig. 1. The resin, to be filled in the tube shall be of strongly basic anion exchange resin of grain size 215 to 150 micron IS sieve (*see* IS 460). Fill about 20 g of the resin with water into the tube and pour 100 ml of ammonium hydroxide (7:100) at a rate of 5 ml per minute, followed by about 50 ml of water at the same rate. Wash the column with 100 ml of hydrochloric acid (1:5) and finally with 50 ml of water. Repeat this procedure.



All dimensions in millimetres.

FIG. 1 ION EXCHANGE RESIN COLUMN

When the resin is used again, only one washing cycle is necessary.

### A-3.3 Reagents

**A-3.3.1** *Concentrated Hydrochloric Acid*

See IS 265.

**A-3.3.2** *Hydrochloric Acid*

1:5 (*v*/*v*).

**A-3.3.3** *Nitric Acid*

1:1 (*v*/*v*)

**A-3.3.4** *Sulphuric Acid*

1:1 and 1:10 (*v*/*v*).

**A-3.3.5** *Acetic Acid*

1:1 (*v*/*v*).

**A-3.3.6** *Ascorbic Acid — Hydrochloric Acid Solution*

Dissolve 0.25 g of ascorbic acid in hydrochloric acid (1:5) and dilute to 100 ml.

**A-3.3.7** *Ammoniacal Eluent*

Dissolve 20 g of ammonium chloride in 1 litre of ammonium hydroxide (7:100).

**A-3.3.8** *Buffer Solution*

Dissolve 250 g of ammonium acetate in water, add 25 ml of glacial acetic acid and dilute with water to 1 litre. Adjust the *p*H value to about 5.5 by means of a *p*H meter.

**A-3.3.9** *Ammonium Fluoride Solution*

5 percent (*m*/*v*).

**A-3.3.10** *Sodium Thiosulphate Solution*

10 percent (*m*/v).

**A-3.3.11** *Xylenol Orange — Indicator Solution*

Dissolve 9.1 g of xylenol orange in 100 ml of 50 percent ethyl alcohol (*v*/*v*).

**A-3.3.12** *Sodium Diethyl Dithiocarbamate*

**A-3.3.13** *Eriochrome Black T Indicator Solution*

Dissolve 0.5 g of Eriochrome black T in 100 ml of ethyl alcohol and add 4 g of hydroxylamine hydrochloride.

**A-3.3.14** *Zinc Standard Solution*

0.05 M. Weigh accurately 3.269 g of zinc into a 500-ml conical flask and add 20 ml of hydrochloric acid to dissolve it. Heat the solution and dilute with water to exactly 1 litre. Standardize this solution with EDTA standard solution (0.05 M) as follows:

> To 10 ml of EDTA standard solution add 180 ml of ammoniacal eluent and a few drops of Eriochrome black T indicator solution and titrate with the zinc solution until the colour changes from blue to violet.

**A-3.3.15** *EDTA Standard Solution*

0.05 M. Dissolve 18.610 g of disodium ethylenediamine tetracetate dihydrate in water and dilute to 1 litre. One millilitre of this solution is equivalent to about 3.269 mg of zinc. Standardize this solution as follows:

> Weigh accurately 0.15 g of zinc into a 500-ml conical flask and add 10 ml of hydrochloric acid to dissolve it. Add 180 ml of ammoniacal eluent neutralize with acetic acid (1:1) and proceed as prescribed in **A-3.4.3**. Calculate the mass of zinc equivalent to 1 ml of EDTA solution as follows:
>
> $$m = \frac{M}{V_1 - V_2}$$
>
> where
>
> $m$ = mass of zinc equivalent to 1 ml of EDTA solution,
>
> $M$ = mass in g of zinc taken,
>
> $V_1$ = volume in ml of EDTA solution consumed in titration for sample, and
>
> $V_2$ = volume in ml of EDTA solution consumed in titration for the blank.
>
> NOTE — For the sample containing small amount of zinc. EDTA solution (0.01 $M$) shall be prepared.

**A-3.4 Procedure**

**A-3.4.1** Weigh accurately 0.3 to 1 g of the sample into a 250-ml beaker. Cover with a watch glass and moisten with a small amount of water.

Add 20 to 30 ml of hydrochloric acid and gently heat to volatilize sulphur contained in sulphide as hydrogen sulphide gas. Then add 10 ml of nitric acid, continue heating to decompose the sample completely. Add 10 ml of sulphuric acid (1:1) and heat to evolve white fumes. After cooling add 50 ml of water, heat to dissolve soluble salts and allow to cool to room temperature. Filter the precipitate and wash several times with cold sulphuric acid (1:10). Collect the filtrate and washings in a 250-ml beaker and pour into the dropping funnel of the apparatus. Add 16 ml of hydrochloric acid and water to make to 100 ml and mix well.

**A-3.4.2** Pour the solution on to the column at a rate of about 6 ml per minute and allow the zinc to be absorbed. After completion of the pouring, add 100 ml of hydrochloric acid followed by 100 ml of ascorbic acid-hydrochloric acid solution and finally 100 ml of hydrochloric acid to wash the column, adding about 7 ml of each solution per minute.

**A-3.4.3** Add 180 ml of the ammoniacal eluent at a rate of about 6 ml per minute to elute the zinc from the column and collect the effluent in a 500-ml beaker. Neutralize the solution with acetic acid and add 20 ml of buffer solution to adjust the *p*H value to 5.0 to 5.5. Then add 2.5 ml of ammonium fluoride solution 5 percent (*m*/*v*), 5 ml of sodium thiosulphate solution [ 10 percent (*m*/*v*)] and a few drops of xylenol orange indicator solution and titrate with EDTA solution until the solution changes colour from pink to yellow.

**A-3.5 Calculation**

$$\text{Zinc (as Zn), percent by mass} = \frac{m\,(V_1 - V_2)}{M} \times 100$$

where

$m$ = mass in g of zinc equivalent to 1 ml of EDTA solution,

$V_1$ = volume in ml of EDTA solution consumed in titration for sample,

$V_2$ = volume in ml of EDTA solution consumed for the blank, and

$M$ = mass in g of the sample taken.

# ANNEX B

## (*Foreword*)

## COMMITTEE COMPOSITION

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(*Continued on page* 7)

(*Continued from page* 6)

## Auxiliary Chemicals Subcommittee, CHD 3:4

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc : No. CHD 3 ( 169 ).

**Amendments Issued Since Publication**

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
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